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TECHLAW INC.

RZ2.R05020.01.FD.239

May 8, 1998

Mr. Brian Freeman
U.S. Environmental Protection Agency
Region 5 DE-9J
77 West Jackson Boulevard
Chicago, IL 60604

Reference: EPA Contract No. 68-W4-0006; Work Assignment No. R05020; Quality Assurance Project Plan Development and Screening; Chemetco, Inc., Hartford, IL; EPA ID No. ILD048843809; Revised Site-Specific Sampling and Analysis Plan; Task 05 Deliverable

Dear Mr. Freeman:

Please find enclosed TechLaw's Revised Site-Specific Field Sampling and Analysis Plan (SAP) for sampling activities proposed for the Chemetco, Inc. (Chemetco) facility located in Hartford, Illinois. This SAP proposes the collection of waste, soil, surface water, and sediment samples necessary to assist U.S. EPA, Region 5 in characterizing potentially hazardous waste streams and potential off-site releases of hazardous constituents at the Chemetco facility.

Please contact me at (312) 345-8963 if you have any questions.

Sincerely,

Patricia Brown-Derocher
Regional Manager

cc: F. Norling, EPA Region 5, w/o attachment
P. Kuefler, EPA Region 5
W. Jordan/Central Files

K. Higgins
Chicago Central Files

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CHEMETCO, INC.
EPA ID NO. ILD048843809

**SITE-SPECIFIC SAMPLING AND ANALYSIS PLAN
WASTE, SOIL, SURFACE WATER,
AND SEDIMENT SAMPLING**

Submitted to:

**Mr. Brian Freeman
U.S. Environmental Protection Agency
Region 5 DE-9J
77 West Jackson Boulevard
Chicago, Illinois 60604**

Submitted By:

**TechLaw, Inc.
20 North Wacker Drive, Suite 1260
Chicago, Illinois 60606**

**EPA Work Assignment No.
Contract Number
Contractor WAM
Contractor WAM Telephone No.
EPA WAM
EPA WAM Telephone No.**

**R05020
68-W4-0006
Patricia Brown-Derocher
312/345-8963
Brian Freeman
312/353-2720**

May 8, 1998

**CHEMETCO, INC.
HARTFORD, ILLINOIS
EPA ID NO. ILD048843809**

**SITE-SPECIFIC SAMPLING AND ANALYSIS PLAN
WASTE, SOIL, SURFACE WATER,
AND SEDIMENT SAMPLING**

The following constitutes the Site-Specific Field Sampling and Analysis Plan (SAP) for waste, soil, surface water and sediment sampling to be performed by TechLaw at the Chemetco, Inc. (Chemetco) facility in Hartford, Illinois. It is anticipated that sampling activities will be undertaken May 28 and 29, 1998. The TechLaw sampling activities will utilize a three person Sampling Team in addition to the U.S. EPA Technical Lead, Mr. Patrick Kuefler, and a representative of the Illinois EPA (IEPA), Mr. Chris Cahnovsky.

This SAP will be used in conjunction with TechLaw's U.S. EPA-approved Region 5 Generic Quality Assurance Project Plan (QAPP) for Sampling Operations, dated January 1995. TechLaw has selected QST Environmental Laboratory (Gainesville, Florida), a TechLaw Team Subcontractor, to perform the analyses required under this SAP.

Purpose and Objective

This SAP has been prepared to allow for the collection and analysis of solid waste streams, soil, surface water and sediment samples at the Chemetco facility. These samples will be collected in support of U.S. EPA Region 5's ongoing enforcement case development activities for this site. These activities relate to the potential improper identification of solid and hazardous waste streams and potential releases of hazardous constituents from the Chemetco facility. A summary of the sampling effort is presented in Table 1, including the identification of sample numbers and Quality Assurance/Quality Control (QA/QC) samples. A summary of analytical methods is presented in Table 2, including recommended sample containers, holding times, and preservatives related to the sampling.

Site Description

The Chemetco facility is located at the intersection of Illinois Route 3 and Oldenberg Road in Madison County, Illinois (Figure 1). The facility is located in a primarily agricultural and light-industrial area. Chemetco operations are conducted on an approximately 40-acre parcel of land surrounded by a chain link fence (Figure 2). Chemetco owns an additional 230 acres of land in the vicinity of the facility.

The Chemetco facility was constructed in 1969 and initiated operations as a copper smelter in 1970 to derive copper and other non-ferrous metals and alloys from recyclable copper bearing scrap and manufacturing residues. The Chemetco facility produces anode copper, cathode copper, and crude lead-tin solder.

The Chemetco facility is located in the floodplain of the Mississippi River in an area locally referred to as the American Bottoms. This area is characterized by relatively flat topography which produces minimal runoff. Precipitation either infiltrates to groundwater or evaporates as the surface gradient of the facility property has been estimated at only 12 inches per mile.

Solid Waste Streams

File material and information obtained from the IEPA has identified four solid waste streams requiring characterization at the Chemetco facility. These waste streams are:

- Waste Slag;
- Baghouse Dust;
- Zinc Oxide; and,
- Spent Refractory Brick.

Waste slag is generated from both water-cooled and air-cooled processes in the Slag Drying and Screening Building. Slag is screened and subsequently stored into five separate slag piles identified as Units (Figure 2).

A total of four baghouses are utilized at the facility to control air emissions, including one baghouse associated directly with the Slag Drying and Screening Operation, one baghouse associated with the Slag Grinding Operations, and a roof baghouse on the Slag Drying and Screening Operation Building. Information obtained from the IEPA indicates that significant volumes of baghouse dust accumulates on concrete and/or asphalt surfaces outside of the Slag Drying and Screening Operation Building. In addition, the IEPA has indicated that baghouse dust has been stored on-site at the Chemetco facility in the slag pile Units (Figure 2).

Process wastewater generated from a venturi scrubber system is currently discharged to an open concrete tank for settling solids which are subsequently de-watered in a zinc oxide filter press. The filter cake from the press is described in file material as zinc oxide. In the past, process wastewater was routed to lagoons for settling and subsequent filter press de-watering. The resulting material was store on-site in a zinc oxide pile. This zinc oxide pile was later converted to a Zinc Oxide Bunker, and is still used for zinc oxide storage in current facility operations. File material indicates that the Zinc Oxide Bunker is located in the vicinity of slag Unit 1 (Figure 2).

Spent refractory brick is generated from the smelting operations and is stored on-site. Information obtained from the IEPA indicates that the facility is currently storing a significant volume of spent refractory brick on the south side of the Zinc Oxide Bunker. To process spent

refractory brick, the facility currently operates an IEPA-air permitted rock crusher. This crusher generates residual brick "fines" and wastewater from air emission control.

Waste Sampling

The goal of the waste sampling effort is to obtain representative samples of the solid waste streams. A summary the waste sampling effort is presented in Table 1, while analytical methods for the waste samples are presented in Table 2.

To characterize the waste slag generated at the facility, samples will be collected from the slag pile Units, the slag accumulation areas associated with the Slag Screening Operation, and the slag "fines" discharged from the Slag Drying and Screening Operation. When sampling the slag pile Units, an attempt will be made to sample various sizes of slag pieces. In addition, information obtained from the IEPA indicates that sampling various sizes of slag may need to be undertaken beneath the Screening Operation conveyors where slag has been segregated. Overall, the determination of sample location within the slag pile Units and in the slag processing areas will be based on field conditions and will be undertaken following the direction of the U.S. EPA Technical Lead.

It is anticipated that all slag samples will be collected using hand augering techniques. However, the sampling of the slag pile Units may require the use of heavy equipment to assist with the vertical characterization. It is anticipated that a minimum of five samples will be collected from each slag pile Unit and a minimum of three samples will be taken in each slag processing area. It is expected that up to a total of approximately 50 samples will be taken for the entire slag sampling effort. All slag samples will be analyzed to determine if the samples exhibit the toxicity characteristics for RCRA metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver) using the Toxicity Characteristic Leaching Procedure (TCLP).

To characterize the dust generated from the four baghouses, a minimum of three samples will be taken from each baghouse. This will include samples taken from the two baghouses associated directly with the Slag Drying and Screening Operation, the baghouse associated with the Slag Grinding Operations, and the roof baghouse on the Slag Drying and Screening Operation Building. In addition, a minimum of three samples will be taken each from the baghouse dust accumulating in the vicinity of the Slag Drying and Screening Operation baghouse and the baghouse dust storage area located in the slag pile Units. All baghouse dust samples will be collected using soil sampling techniques and will be analyzed for RCRA TCLP metals.

To characterize the zinc oxide filter press sludge, a minimum of three samples will be taken from the zinc oxide filter press and a minimum of five samples will be taken from the Zinc Oxide Bunker. In addition, a minimum of five samples will be taken from the zinc oxide product storage area where zinc oxide is accumulated for off-site transport. All zinc oxide samples will be taken using soil sampling techniques and will be analyzed for RCRA TCLP metals.

To characterize the spent refractory brick, a minimum of five samples will be taken from the spent refractory brick pile on the south side of the Zinc Oxide Bunker. In addition, a minimum of five samples will be taken from the residual "fines" from the brick crusher and a minimum of two samples will be taken from the wastewater generated from controlling air emissions from the rock crusher. The refractory brick samples and the residual "fines" from the rock crusher will be analyzed for RCRA TCLP metals, while the rock crusher wastewater will be analyzed for total RCRA metals.

Soil Sampling

Soil samples will be collected to determine if there have been releases of hazardous constituents to soil in the refractory brick waste pile area and waste handling areas. A summary of sample numbers and field/analytical parameters is presented in Table 1, and analytical methods for soil samples are presented in Table 2.

Up to a total of five soil samples will be collected in each area using hand augering techniques. All soil samples will be analyzed for RCRA total metals. Any soil sample exhibiting a RCRA total metal concentration greater than 20 times the regulatory limit for RCRA TCLP metals will then be analyzed for RCRA TCLP metals.

Background soil samples will be collected in an undisturbed area in the vicinity of the Chemetco facility to determine natural soil concentrations of RCRA metals. The background soil samples will be collected in an area which does not appear to have been impacted by potential releases from the Chemetco facility. A minimum of five background soil samples will be collected using hand augering techniques and will be analyzed for RCRA total metals.

Surface Water and Sediment Sampling

Surface water and co-located sediment samples will be collected to determine if there has been releases of hazardous constituents to Long Lake, which is located south of the operations area of the facility (Figures 1 and 2). Surface water and co-located sediment samples will also be taken in the drainage ditch to the north of Containment Area 4, also located south of the operations area (Figure 2). A summary of sample numbers and field/analytical parameters is presented in Table 1, and analytical methods for the samples are presented in Table 2.

It is anticipated that a minimum of five surface water and co-located sediment samples will be taken. The location of the samples will be based on field conditions and will be selected in coordination with the U.S. EPA Technical Lead.

It is anticipated that surface water samples will be collected by submerging sample containers directly into the surface water. Those areas which are accessible only from a distance will be

sampled using a pre-cleaned beaker attached to an extendable aluminum pole. The surface water samples will be analyzed for total RCRA metals and field measurements of pH, conductivity, and temperature will also be performed using appropriately calibrated instruments.

Sediment samples will be collected and analyzed for total RCRA metals. Depending upon surface water depth and sediment compactness, sediment samples will be collected as follows:

- Sediment sample locations covered by less than six inches of surface water will be sampled using a pre-cleaned stainless steel trowel or spoon to transfer the sediment material directly into the sample container.
- Sediment sample locations covered by more than six inches of surface water will be sampled using a hand auger with the sample being transferred directly into the sample container or into a stainless steel bowl prior to placement into a sample container; or
- Sediment sample locations covered by more than six inches of surface water not amenable to hand augering will be sampled using a PVC pipe with a vacuum pump or capping device.

Quality Control Samples

During the collection of waste, soil, surface water and sediment samples at the Chemetco facility, the TechLaw Sampling Team will collect a field duplicate for every 10 samples per matrix collected. In addition, equipment blanks will be collected at a frequency of one for every 10 samples per matrix collected. Depending upon the sample collection technique used, equipment blanks will be collected from the surface water sample beaker, hand auger, and PVC sampling pipe. Duplicates and equipment blanks will be analyzed for the same constituents as the associated samples.

One matrix spike/matrix spike duplicate (MS/MSD) sample will be collected for every 20 samples of each matrix collected. These MS/MSD samples will be analyzed for the same constituents as those in the sample matrix being analyzed.

Laboratory quality control requirements are outlined in the TechLaw's U.S. EPA-approved Region 5 Generic QAPP.

Decontamination

Decontamination of sampling equipment will be undertaken according to TechLaw's U.S. EPA-approved Region 5 Generic QAPP for Sampling Operations. Sampling equipment will be decontaminated with a Alconox® soap wash, a potable water rinse, and a de-ionized water rinse. All decontamination solutions will be collected in a five to 55-gallon container/drum, depending upon the expected volume.

Investigation Derived Waste Management

The sampling activities and potential decontamination procedures outlined in this SAP are not expected to generate significant volumes of Investigative Derived Waste (IDW). It is anticipated that the actual environmental sampling will generate little or no IDW, depending upon the sampling procedures ultimately used according to field observations. As the analytical procedures are limited to inorganics, steps will be taken to ensure that only the volume needed for analysis will be collected. In addition, when applicable, media collected for analysis will be placed directly into the sample containers.

The sampling equipment decontamination solutions will be collected in a five to 55-gallon container/drum, depending upon the expected volume, with disposal subsequently arranged at the request of the U.S. EPA Technical Lead. Appropriate analysis of the IDW will be performed and the wastes managed accordingly.

Sample Collection, Preparation, Custody and Shipment

The samples collected by TechLaw will remain in the custody of the TechLaw Sampling Team until shipment to the analytical laboratory. The sample bottles will be appropriately labeled and tagged with U.S. EPA sample tags in accordance with the TechLaw's U.S. EPA-approved Region 5 Generic QAPP.

A chain-of-custody (COC) form will accompany the samples from the point of origin to the analytical laboratory. When the COC is signed by the laboratory, a copy of the COC will be immediately forwarded by TechLaw to the U.S. EPA Technical Lead for subsequent delivery to Chemetco facility representatives.

The samples will be collected in certified-clean sample containers obtained from QST Environmental Laboratory. All samples collected at the Chemetco facility by the TechLaw Sampling Team will be packaged and shipped to QST Environmental Laboratory (Gainesville, Florida) in accordance with the shipping and custody procedures outlined in TechLaw's U.S. EPA-approved Region 5 Generic QAPP.

Analytical Requirements

Analytical and QA/QC requirements, including calibration procedures and frequencies, are outlined in the TechLaw's U.S. EPA-approved Region 5 Generic QAPP. The analytical methods, containers, preservatives, and holding time requirements are presented in Table 2.

Data Validation

Analytical data will be generated by the subcontractor laboratory and provided to TechLaw in conformance with CLP-like reporting protocols. The resulting data will undergo a 100 percent data validation effort by a member of the TechLaw Team, independent of the sampling team. This validation will be in conformance with the Functional Guidelines for Inorganic Data Validation. Specific data package and data validation requirements are outlined in the U.S. EPA-approved, TechLaw Generic QAPP.

Project Schedule and Report Deliverables

The sampling activities are planned for May 28-29, 1998 with mobilization to the facility occurring on May 27, 1998. A data validation report will be generated within 21 days of receiving the laboratory data package for the final analysis. Within 14 days of the receipt of the data validation report, a final sampling report will be prepared and submitted to the U.S. EPA Work Assignment Manager (EWAM) and the U.S. EPA Technical Lead (Mr. Kuefler). The report will include a discussion and statistical analysis of the results of the sampling effort. In addition, the report will describe the sampling locations and techniques, any problems that were encountered, deviations from this SAP, and any other observations, including photographs, made during the sampling activities.

Project Organization

The EWAM for this project is Mr. Brian Freeman, and the U.S. EPA Technical Lead is Mr. Kuefler. Mr. Cahnovsky of the IEPA is the State of Illinois representative for the project.

The TechLaw Work Assignment Manager (TWAM) for this project is Ms. Patricia Brown-Derocher, and the TechLaw Technical Lead for this project is Mr. Kevin Higgins. TechLaw field sampling personnel will include Mr. Higgins (Team Leader), Mr. Douglas Updike, and Mr. Michael Powers. Mr. Powers will also serve as TechLaw's Site Safety Officer.

The laboratory for this project is QST Environmental Laboratory (Gainesville, Florida). Data validation will be performed by appropriately qualified members of the TechLaw Team.

TABLE 1
SAMPLE COLLECTION SUMMARY

**TABLE 1
SAMPLE COLLECTION SUMMARY**

Waste Stream/Area of Concern		Matrix	Number of Samples	Sample Depths	Field Parameters	Analytical Parameters	Equipment Blanks (EB)	Field Duplicates (FD)	MS/MSDs
Waste Slag		Waste	25 - 50	Based on Field Observations	None	TCLP Metals	3 - 6	3 - 6	2 - 3
Baghouse Dust		Waste	18	Based on Field Observations	None	TCLP Metals	2	2	1
Zinc Oxide	Filter Press	Waste	3	Based on Field Observations	None	TCLP Metals	1 - 2	1 - 2	1
	Bunker	Waste	5						
	Product Storage	Waste	5						
Spent Refractory Brick	Brick Pile	Waste	5	Based on Field Observations	None	TCLP Metals	1	1	1
	Crusher Fines	Waste	5	Based on Field Observations	None	TCLP Metals			
	Crusher Wastewater	Wastewater	2	Based on Field Observations	None	Total RCRA Metals	1	1	1
Refractory Brick Storage Area		Soil	5	0 - 6 Inches	None	Total RCRA Metals*	1	1	1
Waste Handling Areas		Soil	5	0 - 6 Inches	None	Total RCRA Metals*			
Background Area		Soil	5	0 - 6 Inches	None	Total RCRA Metals	1	1	1
Surface Water		Surface Water	5	Surface	pH, Conductivity, Temperature	Total RCRA Metals	1	1	1
Sediment		Sediment	4	0 - 6 Inches	None	Total RCRA Metals	1	1	1
Totals MS/MSDs: 10 - 13			Samples: 92 - 142			EBs: 12 - 16 FDs: 12 - 16			

* Any soil sample exhibiting a RCRA total metal concentration greater than 20 times the regulatory limit for RCRA TCLP metal concentration will also be analyzed for RCRA TCLP metals.

TABLE 2

**ANALYTICAL METHODS, SAMPLE CONTAINERS,
PRESERVATIONS, AND HOLDING TIME REQUIREMENTS**

TABLE 2

**ANALYTICAL METHODS, SAMPLE CONTAINERS,
PRESERVATIONS, AND HOLDING TIME REQUIREMENTS**

Parameters	Analytical Method	Matrix	Holding Time	Container	Preservative
TCLP Metals	Sample Preparation: SW-846 Method 1311 Sample Analysis: SW-846 Method 6010B	Waste, Soil	6 months*	4-ounce glass jar	Cool to 4°C
Total RCRA Metals	Sample Preparation: SW-846 Method 3010/3005 (Water) 3050 (Soil/Sediment) Sample Analysis: SW-846 Method 6010B and 7000 Series as necessary based on results for arsenic, lead, and selenium	Surface Water, Wastewater	6 months*	1-liter poly bottle	HNO ₃ to pH < 2, Cool to 4°C
		Soil, Sediment	6 months*	8-ounce glass jar	Cool to 4°C

* Holding Time for total mercury is 28 days.

FIGURE 1
FACILITY LOCATION

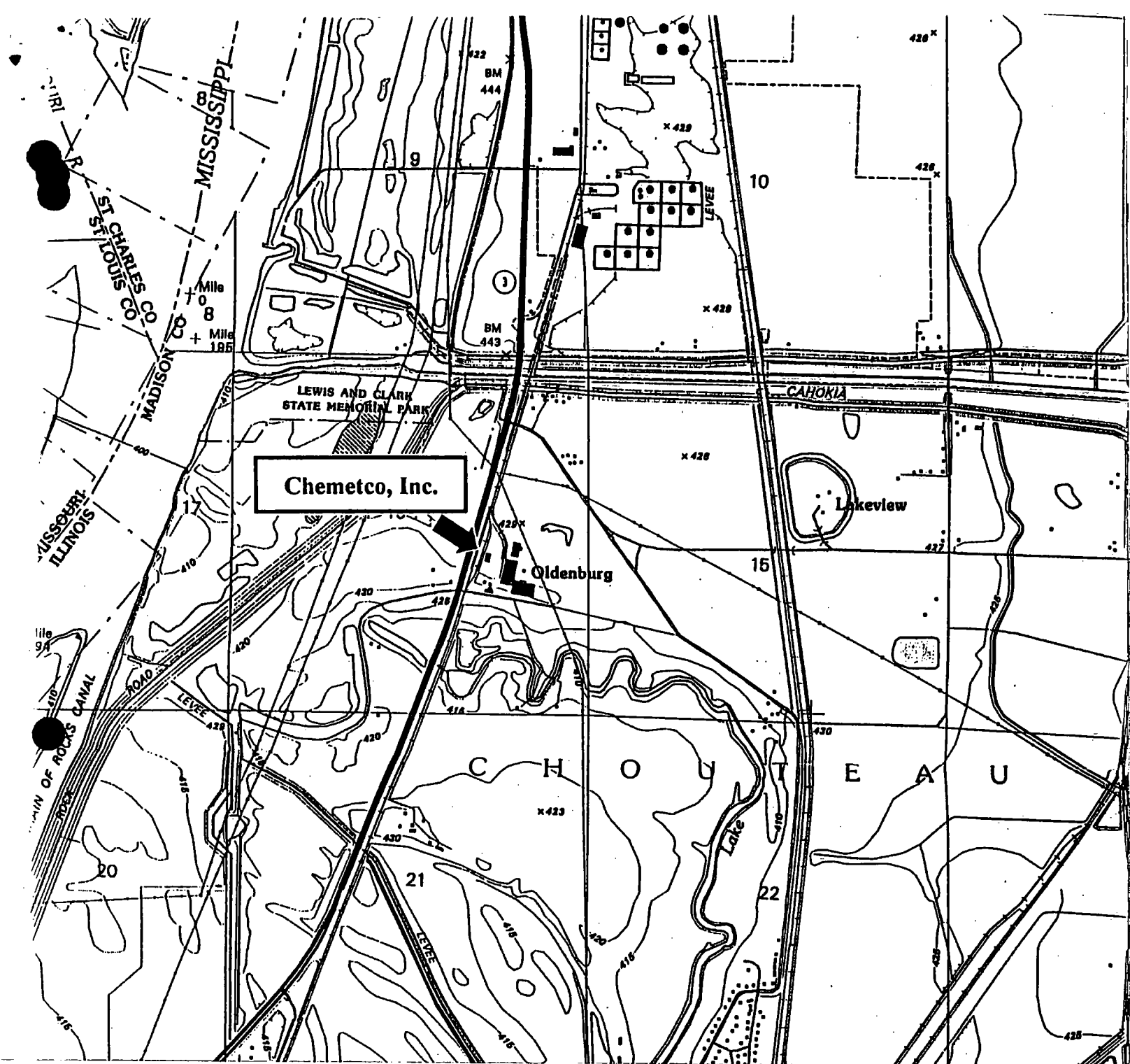
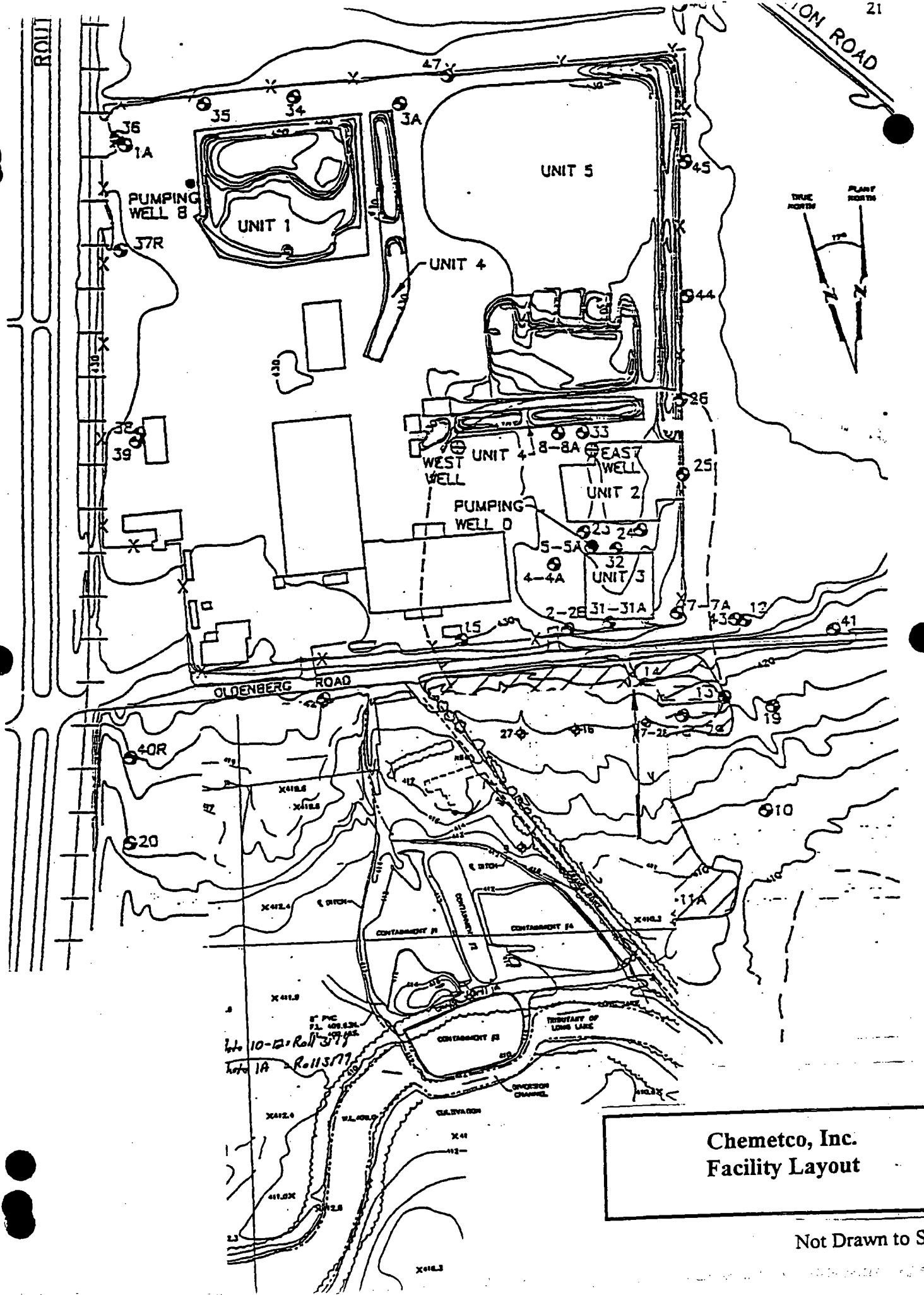


FIGURE 2
FACILITY LAYOUT



Chemetco, Inc.
Facility Layout

Not Drawn to Scale

cc: D Payne

**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V**

DATE: FEB 19 1988

SUBJECT: L.C. Metals Analysis of EP Extracts for Chemetco, Inc.

FROM: Curtis Ross, Director
Central Regional Laboratory

TO: William H. Miner, Acting Chief
Solid Waste Branch

Attn: Kevin Pierard

David A. Payne, Chemist, Central Regional Laboratory (CRL), has reviewed the information from Chemetco, Inc. dated December 28, 1987, that we received on January 25, 1988. The information is a copy of analysis records, for cadmium and lead EP toxicity testing of a representative solid sample, by L.C. Metals Laboratory. The records are for analytical operations performed on December 10, 12, and 15, 1987 and are intended to demonstrate certain deficiencies identified in the CRL's December 8, 1987 evaluation of L.C. Metals, and in the CRL's December 22, 1987 evaluation report, have been corrected. The December 22nd recommendations were orally transmitted to L.C. Metals and Chemetco staff on December 8 by Mr. Payne. You transmitted a summary of them in writing, to Chemetco on January 11, 1988 as part of a QAPP disapproval (see page 3 of your January 11th letter).

We have reviewed the most recent Chemetco submission that you sent, on February 2, 1988 to our Division's Quality Assurance Section. We had ceased review of Chemetco's December 28th submission, awaiting this new information; however the February 2nd material deals only with QAPP issues and not with the December 8, 1987 laboratory evaluation. Our review of the Chemetco December 28, 1987 information follows.

A. Analysis records of December 10, 1987 are titled "EP Toxicity Worksheet" and provide:

1. Use of the stirring extractor and automated pH control.
2. A pH calibration check at the end of a 24 hour extraction time period. A pH 7 buffer was used for this check of calibration accuracy. It is suggested a pH 4.0 buffer is better than a pH 7 buffer, because pH 4.0 is nears the pH 4.5 value of interest. This was orally transmitted to L.C. Metals.
3. Strip chart records of measured pH values during the extraction. These records were in effect on December 8, 1987.

This corrects the deficiencies cited in recommendations #2 and #4a. of our December 22, 1987 memo report.

B. Analysis records of December 12, 1987, titled "Sample Preparation Report (EP Toxicity Extract Preparation)", provide:

1. A sample digestion log for one sample blank and 5 different sample extracts. One of these 5 samples is used for the analysis records of December 10th and 15th.

This corrects the deficiency cited in Recommendation #4b of our December 22, 1987 memo report.

C. The analysis records of December 15, 1987 provide:

1. Atomic absorption absorbance values as a function of cadmium or lead concentrations (4 or more in number) for
 - a. instrument calibration curves
 - b. a reagent blank of 12/12/87 with method of standard addition calculations.
 - c. Sample "C reprep" (of 12/10/87 and 12/12/87) analysis with method of standard addition calculations.
 - d. Control solution (of 12/12/87 containing 1.0 mg/lCd and 5.0 mg/lPb) analysis with method of standard additions calculations.
 - e. Instrument calibration checks.
2. All calibration curves, and method of standard additions for blanks, as being linear with linear regression coefficients > 0.995 . Intercepts do not significantly deviate from zero concentration.
3. All method of standard additions to sample EP toxicity extracts being linear with regression coefficients > 0.995 .
4. Control solution values, calculated in the same way as sample extracts, have a measured cadmium concentration of 1.09 mg/l versus a "true value of 1.0 mg/l, and a measured lead concentration of 4.89 mg/l versus 5.0 mg/l. These measurements of an independently prepared reference solution are of excellent accuracy.
5. Calibration check solution values being measured as 1.02 mg/l cadmium and 5.17 mg/l lead, versus 1.0 and 5.0 mg/l values from their initial calibration curves.

These items correct deficiencies cited in Recommendation #4d. of our December 22, 1987 memo report. Other recommendations, concerning appropriate use of method of standard calculations, within the text of this December 22nd report, were also incorporated in the analyses of December 15, 1987.

D. Conclusions

L.C. Metals provided accurate and appropriate measurements of cadmium and lead in EP Extracts. L.C. Metals should be considered acceptable for these measurements.

cc: D. Payne, CRL
A. Jirka, DQAB
M. Long, DQAB

U.S. Environmental Protection Agency
Environmental Monitoring and Support Laboratory - Cincinnati
QUALITY CONTROL SAMPLES

Instructions for EP EXTRACT METALS Analyses

CAUTION: Read Instructions Carefully Before Opening Ampuls.

The requested set(s) of EP Extract Metals are enclosed in this package. These quality control samples were designed to be used with the Extraction Procedure (EP) given in USEPA Manual "Test Methods for Evaluating Solid Waste", SW-846, July 1982. The EP is designed to simulate the leaching a waste will undergo if disposed of in a sanitary landfill only. It is a laboratory test in which a representative sample of waste is extracted with distilled water maintained at a pH of 5 using acetic acid. The extract obtained from the EP Extract is then analyzed to determine if any of the thresholds established for the eight elements have been exceeded. The quality control sample ampul identified as Concentrate #1 contains Ba, Cd, Cr, Pb, Hg and Ag. The quality control sample ampul identified as Concentrate #2 contains As and Se.

SAMPLE PREPARATION

The quality control samples are to added to laboratory pure water. The laboratory pure water represents the instances where the wastes are less than 0.5% solids and hence are not processed through the entire Extraction of Solid Materials section as stated in the USEPA Manual.

Stabilize the temperature of the ampul at 20°C. When ready to begin the analysis, open the ampul by snapping off the top at the break area on the neck. Transfer exactly 10.0 mL of the concentrate to a 1000 mL volumetric flask. Bring to volume with laboratory pure water. The sample is now ready for analyses.

A sheet containing the statement of added levels is attached with these instructions for use as you desire. If there are any questions or problems, please contact:

Quality Assurance Branch
Environmental Monitoring and Support Laboratory
U.S. Environmental Protection Agency
Cincinnati, OH 45268

U.S. Environmental Protection Agency
Environmental Monitoring and Support Laboratory - Cincinnati

QUALITY CONTROL SAMPLES

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SAMPLE PREPARATION

The quality control samples are to added to laboratory pure water. The laboratory pure water represents the instances where the wastes are less than 0.5% solids and hence are not processed through the entire Extraction of Solid Materials section as stated in the USEPA Manual.

Stabilize the temperature of the ampul at 20°C. When ready to begin the analysis, open the ampul by snapping off the top at the break area on the neck. Transfer exactly 10.0 mL of the concentrate to a 1000 mL volumetric flask. Bring to volume with laboratory pure water. The sample is now ready for analyses.

A sheet containing the statement of added levels is attached with these instructions for use as you desire. If there are any questions or problems, please contact:

Quality Assurance Branch
Environmental Monitoring and Support Laboratory
U.S. Environmental Protection Agency
Cincinnati, OH 45268

**U.S. Environmental Protection Agency
Environmental Monitoring and Support Laboratory - Cincinnati**

EP EXTRACT METALS - Quality Control Samples

TRUE VALUES

When diluted to volume according to the instructions, the samples contain the following element expressed as mg/liter. The true value represents the actual weighings and all subsequent dilutions. The mean recovery (\bar{X}) and the standard deviation (S) were developed from the data received from the referee laboratories. The 95% confidence interval represents the mean recovery plus or minus 2.086 standard deviations.

Element	Concentration	True Value	\bar{X}	S	95% Confidence Interval
Ba	1	100	97.8	10.2	76.5 - 119
Cd	1	1.00	1.01	0.03	0.95 - 1.07
Cr	1	5.00	5.06	0.42	4.18 - 5.94
Pb	1	5.00	5.12	0.44	4.20 - 6.04
Hg	1	0.20	0.17	0.04	0.08 - 0.26
Ag	1	5.00	4.97	0.28	4.39 - 5.55
As	2	5.00	4.97	0.70	3.51 - 6.43
Se	2	0.99	0.94	0.15	0.64 - 1.25

**U.S. Environmental Protection Agency
Environmental Monitoring and Support Laboratory - Cincinnati**

QUALITY CONTROL SAMPLES

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SAMPLE PREPARATION

The quality control samples are to added to laboratory pure water. The laboratory pure water represents the instances where the wastes are less than 0.5% solids and hence are not processed through the entire Extraction of Solid Materials section as stated in the USEPA Manual.

Stabilize the temperature of the ampul at 20°C. When ready to begin the analysis, open the ampul by snapping off the top at the break area on the neck. Transfer exactly 10.0 mL of the concentrate to a 1000 mL volumetric flask. Bring to volume with laboratory pure water. The sample is now ready for analyses.

A sheet containing the statement of added levels is attached with these instructions for use as you desire. If there are any questions or problems, please contact:

Quality Assurance Branch
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Cincinnati, OH 45268

**U.S. Environmental Protection Agency
Environmental Monitoring and Support Laboratory - Cincinnati**

EP EXTRACT METALS - Quality Control Samples

TRUE VALUES

When diluted to volume according to the instructions, the samples contain the following element expressed as mg/liter. The true value represents the actual weighings and all subsequent dilutions. The mean recovery (\bar{X}) and the standard deviation (S) were developed from the data received from the referee laboratories. The 95% confidence interval represents the mean recovery plus or minus 2.086 standard deviations.

Element	Concentration	True Value	\bar{X}	S	95% Confidence Interval
Ba	1	100	97.8	10.2	76.5 - 119
Cd	1	1.00	1.01	0.03	0.95 - 1.07
Cr	1	5.00	5.06	0.42	4.18 - 5.94
Pb	1	5.00	5.12	0.44	4.20 - 6.04
Hg	1	0.20	0.17	0.04	0.08 - 0.26
Ag	1	5.00	4.97	0.28	4.39 - 5.55
As	2	5.00	4.97	0.70	3.51 - 6.43
Se	2	0.99	0.94	0.15	0.64 - 1.25

U.S. Environmental Protection Agency
Environmental Monitoring and Support Laboratory - Cincinnati

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Quality Assurance Program
Environmental Monitoring and Support Laboratory
U.S. Environmental Protection Agency
Cincinnati, OH 45260



JACOBS ENGINEERING GROUP INC.
ENVIRONMENTAL SYSTEMS DIVISION

March 11, 1987

Mr. Kevin Pierard
U.S. Environmental Protection Agency
RCRA Enforcement
540 South Dearborn Street
Chicago, IL 60604

Re: Work Assignment #102

Dear Mr. Pierard:

Please find submitted herewith a draft of the Site Specific Sampling and Analysis Quality Assurance Project Plan for the special study at Chemetco, Inc., Hartford, Illinois. Four additional copies of this draft are being prepared by Metcalf and Eddy and will be submitted by March 13, 1987. A final Sampling Plan and QAPP will be prepared upon receipt of comments from U.S. EPA.

If you have any questions or require additional information, please feel free to contact me at (312) 806-9119.

Sincerely,

Dean Geers
Regional Manager

enclosure

cc: A. Kaganove, U.S. EPA
B. Goodwin, Metcalf & Eddy
R. Williams, Jacobs Engineering

C.C. D. Payne, CR



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5

230 SOUTH DEARBORN ST.

CHICAGO, ILLINOIS 60604

AUG 14 1990

REPLY TO THE ATTENTION OF:

MEMORANDUM

5SCR

SUBJECT: Laboratory Evaluation of Northern Laboratories, Valparaiso Indiana for Quemetco, Indianapolis, Indiana RFI

FROM: Charles T. Elly, Acting Director
Central Regional Laboratory

Chuck E. Elly

TO: William Muno, Chief
RCRA Enforcement Branch

ATTN: Walt Francis

Our ESAT contractor evaluated Northern Laboratories on July 25, 1990, for the Quemetco RFI. We agree with the recommendations and conclusions of the attached evaluation report in the following:

1. The QAPP should identify the sample preparation and analytical methods to use. The attached report identifies correct method citations from RCRA SW-846.
2. Soil preparation procedures/test procedures should be identified for alkalinity and sulfate. Northern Laboratories will need to decide if their MTB Auto Analyzer method for sulfate is acceptable in the presence of CaCl_2 or LiCl extracting reagent for soil, or if the manual turbidimetric procedure will be required. Northern Laboratories has a little bit of work to do for their sulfate in soil if the auto analyzer method is to be used.
3. Northern Laboratories has agreed to determine tin by ICP emission spectroscopy instead of flame atomic absorption as currently specified by the draft QAPP. Northern Laboratories will need to upgrade their methodology for this element by obtaining a new standard solution, and by implementing interelemental correction (IEC) factors and an instrument detection limit (IDL). This is expected to be done relatively easy.

4. Lead, at relatively high concentration, will be tested by ICP emission spectroscopy instead of by the more tedious graphite furnace technique designed for concentrations between 5 and 200ug/l. The emission spectroscopy method is most applicable to the high lead concentrations expected in soils.
5. With the advent of the new TCLP regulations, we expect Northern Laboratories to use this extraction technique instead of EP Toxicity. We expect no problems in implementing the TCLP for metals, since Northern Laboratories has performed well for EP Toxicity in the past.
6. The ESAT team member did review Northern Laboratories performance for general chemistry (sulfate, alkalinity, chloride) in relation to specific work Northern Laboratories did for Region V as Special Analytical Services (SAS) for the Superfund Contract Laboratory Program. Attention was drawn to the completeness and attention to detail for this work. Northern Laboratories needs to assure that QC details specified in the QAPP will be followed for general chemistry.
7. While it is not so stated in the attached report, Northern Laboratories performs metals analysis acceptably as part of Superfund's Contract Laboratory Program. Testing procedures specified for metals in the QAPP are equivalent to the CLP procedures. Tin is not a CLP parameter, so this is why the report has specifically evaluated this element.

We and ESAT concur that Northern Laboratories is appropriate and acceptable for the Quemetco RFI as soon as they implement or correct the items identified above through documentation of the finalized QAPP for the project.

Two other items were discussed during the laboratory evaluation.

1. Thallium analysis is not part of the proposed RFI. It can readily be done by graphite furnace atomic absorption determinations as is now done by Northern Laboratories for the Superfund CLP. Our previous conversations with Quemetco/Canonie did not mandate the determination of thallium, although thallium is an Appendix IX metal. It would be desirable to do for completeness, but we may not want to delay the project if Canonie does not want to do it.

2. A Cation Exchange Capacity (CEC) method or Superfund SAS procedure was provided to Northern Laboratories (see attached copy) for informational purposes. SW-846 Method 9081 is another alternative to use. Northern Laboratories will have to decide the procedure to use and so specify in the QAPP. "Methods of Soil Analysis" discusses the utility of several CEC procedures used for soils. Northern Laboratories has this book of standard methods for soil analysis.

cc. G. Schupp, QAS
D. Payne, CRL ✓

July 26, 1990

TID: 0590-0630

To: Dave Payne

Task #: 3073/3299

FROM: Duane Kruse

DKruse P. Herley

Sung: Audit of Northern Laboratories for the RCRA Facility Investigation of Quemetco, Inc.

On July 25, 1990, I audited Northern laboratories' ability to perform the analytical work as outlined in the QAPP for Quemetco, Inc. The QAPP was prepared by Canonie Environmental. The following people were involved in the audit process:

Dr. El Nigar	President NLE	
Dr. Stephen Havlick	Vice-President	Coast to Coast Analytical
Glen Gorski	Project Mgr	Canonie
Terence Ashworth	Project Scientist	Canonie
Harold McCarron	QA/QC	NLE
Elizabeth Janssen	CLP Mgr	NLE
Adrienne Byrnes	Analytical Mgr	NLE

The first meeting was primarily a discussion of the QAPP and inconsistencies that were found during review. The following subjects were discussed.

I asked them to consider the need for including Tl in the list of analytes. Canonie will contact a geologist about Tl as a contaminate in Pb.

Canonie agreed that the total solids method must be included for solid samples. I also indicated that any samples that are air dried will also need to have total solids determined. All solid sample results must be calculated on a dry weight basis.

The QAPP listed method 7420 for Hg on solid samples. The correct method is 7421 and the method includes the sample preparation. The detection limit for Hg must be changed from 1 ug/l to 0.2 ug/l. Canonie agreed to these changes. NLE also indicated that their routine detection limit is 0.2 ug/l.

It was also agreed that the option of analyzing samples for Pb by ICP would be allowed. I emphasized that they would need to state the level above which ICP would be used.

A water method for sulfate was specified for soil samples without any preparation method. I recommended that either a calcium

chloride or lithium chloride extraction must be used to prepare the soil samples. Calcium ion is an interferant in the automated sulfate procedure. I further recommended that the turbidimetric method may have to be used. NLE would like to use the automated method. My position was that this is acceptable only if they can demonstrate that Ca and Li interference can be controlled or eliminated. They should try the method with soil or insert NBS reference material.

The alkalinity/acidity test methods also did not include a preparation method for soils. This was also discussed. It was agreed that preparation methods for alkalinity/acidity and sulfate will be developed and included in the QAPP. I provided copies of Region 5 SASs for these parameters as guidelines for their method development.

The preparation for water samples for Hg and As analyses was incorrectly listed as 3005. This will be corrected.

Sn will be analyzed by ICP. IDLs and IEC factors will be determined before the start of the project.

The quality control objectives for this project were discussed. Use of a 20% RPD limit for precision in all samples is inappropriate for low levels and for soils. For low levels they will allow a duplicate difference set at a value for each analyte. This may be the numerical equivalent of CRDL from the CLP statement of work 7/88. For soils the limit will be 35% RPD or 2 x the water limit for low levels. QC limits must also be established for all wet chemistry and TCLP. I recommended that a table should be added to the QAPP that outlines all QC limits.

Data validation was not covered by the QAPP. Canonie and NLE will decide who will validate the data and what methods are to be used. The 7/88 guidelines will be sent to Harold McCarron.

NLE asked about sub-contracting the CEC test. I had thought that CEC was not to be performed by NLE as per the QAPP. NLE was seeking guidance as to which method to use. I indicated that I would send a copy of the Region 5 SAS for CEC.

NLE showed me the latest results for the analysis of WP024, concentrations 1 and 2. All parameter relevant to the QAPP were acceptable.

Two SAS cases similar to the packages that will be provided for the QAPP were made available for inspection. The report forms were modified CLP forms. This format is acceptable, but the lab will need to concentrate on the completeness of the case. Sulfate data for calibration blanks and calibration verifications were not included on the forms in one case. Titrant standardization data for alkalinity was omitted from another case. NLE agreed that these were problems and that they would concentrate on the completeness of future packages.

The case files will be kept in a file cabinet that is also used for CLP cases. The file cabinet cannot be locked. If this is acceptable for CLP then it is acceptable for this QAPP.

The SOPs for chloride, sulfate and other wet chemistry parameters were inspected. As previously indicated, the SOPs for sulfate and alkalinity/acidity will be rewritten to include soil preparation methods.

An inspection of the lab facilities was also conducted. Temperature logs are attached to the storage cooler and drying ovens. Excursions outside the acceptable temperature range were noted but no corrective actions were documented.

A new deionized water system has been installed which should help solve previous problems with preparation blanks.

An inspection of the ICP revealed that there were no interelement correction factors for Pb or Sn. IECs may not be needed for high levels of Pb but this should be reconfirmed. IECs, IDL and background correction points for tin must be established. The stock standard solution for tin expires next month. New stock will be needed before the project starts.

Sample check-in procedures are adequate. After samples are logged in, the paperwork will be transmitted to the Project Manager. The Project Manager will assign the Analyst to the specific samples to assure that methods specific for the QAPP are used rather than "generic" methods.

The laboratory has a good supply of check sample ampules from the USEPA. They indicated that these are becoming difficult to obtain. I recommended that they should investigate commercial and NBS sources to avoid running out of critical check samples in the middle of a project.

To summarize, Canonie and NLE will make the following general corrections for the Quemetco Project.

1. Include the correct preparation and analytical methods in the QAPP.
2. Tin will be run by ICP after IECs, IDL and background correction points are established.
3. Lead may be run by ICP if the concentration is high enough. The concentration at which ICP is acceptable will be stated in the QAPP.
4. QA/QC limits will be established for all non metal parameters, including TCLP. Matrix specific and concentration specific QC will be documented in the QAPP.

5. Soil preparation methods for sulfate and alkalinity/acidity will be written and included on the QAPP.
6. The Region V SAS for CEC will be sent to NLE for method development or subcontracting.
7. NLE will improve the completeness and attention to detail in the reporting of SAS type parameters.

HC 8a
Andrea Jirka
Attn: M. Long

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

DATE: DEC 22 1987

SUBJECT: Laboratory Evaluation - L.C. Metals Laboratory for EP Toxicity Testing
at the Chemetco, Inc. Site

FROM: Curtis Ross, Director
Central Regional Laboratory - 5SCRL

TO: William H. Miner, Acting Chief
Solid Waste Branch

ATTN: Kevin Pierard

I. INTRODUCTION

On December 8, 1987, David A. Payne, Chemist, Central Regional Laboratory, Region V, EPA visited the L.C. Metals, Inc. laboratory, Granite City, Illinois, to evaluate their EP Toxicity testing procedure for cadmium and for lead. The evaluation was done at the request of the Solid Waste Branch, Waste Management Division, Region V for the RCRA program. L.C. Metals, Inc., is to test 20 or more slag samples (resulting from secondary copper smelting) from Chemetco, Hartford, Illinois. The samples will be from Chemetco's historical slug repository. One hundred gram sample aliquots will be selected from the repository jointly by Chemetco and by U.S. EPA/Illinois EPA representatives. The analysis aliquots will be received by L.C. Metals for EP Extract testing and will require no additional grinding, sewing, etc. Chemetco already has done these preparation steps.

The visit evaluated L.C. Metals' capability for the EP Toxicity Test (cadmium and lead) and made recommendations for certain corrective actions. The recommendations were orally transmitted to L.C. Metals and Chemetco personnel at the time of the evaluation.

II. SUMMARY

1. L.C. Metals laboratory has the capability to acceptably perform the EP Toxicity Test for cadmium and lead. One extraction per day can be done with available extraction equipment.
2. L.C. Metals uses one of the available EP Extraction Test options of Method 1310, SW-846. This option provides for automatic addition of acetic acid to the extraction to maintain the pH value between pH 5.0 and pH 5.2. Metals concentrations (especially lead) can expected to be smaller, by this option, than the metals concentrations determined by federal and State regulatory agencies commonly using a different option of Method 1310. Details are discussed in the attached report.

III. RECOMMENDATIONS

1. Do not split samples for QA purposes. L.C. Metals should obtain smaller concentrations than other laboratories.
2. L.C. Metals must determine the calibration accuracy of their pH controller at the end of each 24 hour, EP Extraction time period, or more often if they desire.
3. After each extraction, sample extracts should be acidified with 2-5 mls nitric acid (pH<2) for the best available preservation. This is not specifically mentioned in Method 1310, SW-846.
4. L.C. Metals should document the following items in their analysis records:
 - a. pH calibration check of pH controller at end of 24 hour extraction time period, or more often.
 - b. Sample digestion log.
 - c. Any background corrections provided for atomic absorption measurements.
 - d. Calibration check standards and instrument blanks as part of the instrument re-zeroing.
5. L.C. Metals should edit past data, for acceptability, prior to formal establishment of control limits for the analysis of control solutions.
6. The above items should be implemented prior to testing of Chemetco slag samples. The above items were orally discussed with L.C. Metals and Chemetco at time of the lab visit.
7. Correction of the above deficiencies can best be demonstrated by L.C. Metals and Chemetco providing the Solid Waste Branch, Region V with a copy of an analysis record for EP Toxicity testing subsequent to the laboratory evaluation.

IV. PERSONNEL

Ms. Jane Drumhiller, Supervisory Chemist, L.C. Metals, was found familiar with the details of the EP Toxicity test. Her education (Analytical Chemistry, M.S.) and experience (at least 3 years employment with L.C. Metals) are sufficient qualifications for the proposed EP Toxicity testing, which will be under her close supervision.

Ms. Susan Schneider, Laboratory Technician, L.C. Metals was not available during the laboratory visit.

V. PHYSICAL FACILITIES

The laboratory physical facilities are principally used for analytical services to L.C. Metals' manufacturing operations. The EP Toxicity testing will require a small percentage of available space. Bench space is adequate for the EP Extraction. Reagent water from a Corning Megapure still is available. Hoods are available for acid digestions. The atomic absorption spectrophotometer used for metal measurements is located in a separate manufacturing building area having adequate space, utilities, and clean atmosphere. Physical Facilities are acceptable for the proposed project.

VI. ANALYTICAL EQUIPMENT AND INSTRUMENTATION

The following equipment and instrumentation for the EP Toxicity test were observed and evaluated. This is acceptable.

A. Extractor and pH Controller

L.C. Metals laboratory has one extractor and pH controller set-up. The extractor is shown in Figure 1 on p. 1310-2 of Method 1310 (SW-846, 3rd ed.) with one modification - The stirring bar is at the bottom of extraction container.

The bar is commonly rotated at 185 RPM during the extraction step. The Chemtrix pH controller, described in Section 7.13 (p. 1310-11) of Method 1310 (SW-846, 3rd ed.), is used to control the pH of the extraction. L.C. Metals laboratory operates the pH controller between pH 5.0 and 5.2. This is in accordance with specifications of Method 1310 in that the extraction pH is to be pH 5.0 + 0.2. The pH controller adds acetic acid when the pH value rises to 5.2 and continues to add acetic acid until the pH value decreases to pH 5.0. A strip chart record of pH is available for each extraction. By definition the pH never decreases below 5.0.

1. Observation: The above extractor meets the requirements and specifications of Method 1310; however, do not expect comparable results between the above method option and the EP extraction options used by regulatory agencies such as Illinois EPA and U.S. EPA. The regulatory agency laboratories will extract several samples at one time commonly using stirring apparatus or the tumbling extractions of Figure 2 and 3 of Method 1310. These extractions require manual adjustments of pH which are expected to decrease to pH 4.8 (or less) for instantaneous values. The minimum pH value may well have a significant effect on the maximum lead concentrations observed for the test.

Recommendation: Expect that L.C. Metals laboratory and Chemetco to obtain smaller EP Extract metal concentrations than regulatory federal and State agencies of Region V, especially for lead. Do not split samples for QA purposes. Consider the EP Extraction apparatus of L.C. Metals to meet requirements of the EP Extraction test.

2. Deficiency: The initial pH value of the EP extraction is measured and recorded and the pH electrode is calibrated at initiation of the test. L.C. Metals does not check the calibration accuracy of the pH electrode at the end of the 24 hour extraction time (This is not a specific requirement of Method 1310, but is an understood requirement). We do not know for sure if there is any drift in pH calibration during 24 hours.

Recommendation: At the end of the 24 hour extraction period, or more often measure the pH value of a standard reference buffer such as the pH 4.01 buffer. L.C. Metals should then record this value. If the measurement error is more than pH 0.10 or if the measurement error is such that the extraction specifications of pH 5.0 ± 0.2 are not met, the extraction must be repeated. At the time of the lab evaluation, L.C. Metals and Chemetco personnel agreed to this requirement.

B. Filtration Apparatus

The laboratory first vacuum filters the EP extraction with a pre-filter to remove most of the solids and secondly a final vacuum filtration using a Gelman 0.45u membrane filter. The filtrate not used for cadmium and lead analysis is archived for any needed reanalysis. This filtration meets the specifications of Method 1310.

C. Atomic Absorption Spectrophotometer

A Perkin-Elmer Model 2380 atomic absorption spectrophotometer (3 years age), equipped with 1) air/acetylene flame, 2) direct concentration read-out and scale-expansion (not used), and 3) printer is available. The instrument does not utilize background correction when using wavelength measurements below 300nm. The instrument is currently being used in an absorbance read-out mode for EP Toxicity measurements.

1. Observation: Background correction is not utilized, as specified by Methods 7130 and 7420 of SW-846, 2nd ed., or 3rd ed. Without background correction of non-specific absorption, cadmium results may be biased 0.01 or 0.02 mg/l high, and lead results may be biased 0.1 or 0.2 mg/l high for EP Toxicity extracts. The method of standard addition calculations used for quantitation of metals will not compensate or correct any background.

Recommendation: Accept the small positive errors due to no background correction. This is to the detriment of Chemetco. If L.C. Metals wishes to correct for background, they can use current hollow cathode lamps (with separate measurements) to provide non-absorbing lines close in wave-length to the 222.8nm cadmium line or to the 283.3nm lead line.

2. Observation: The above atomic absorption instrument is acceptable for compliance determinations of cadmium, lead, and silver in EP Extracts. The instrument is not acceptable for barium and chromium unless the air/acetylene flame is replaced by a nitrous oxide/acetylene flame. Arsenic, mercury, and selenium, require different methodologies or instrumentation.

VII. SAMPLE COLLECTION, HANDLING, AND PRESERVATION

Solid samples received by L.C. Metals from Chemtco are documented on the Chain-of-Custody record provided by the November 1987 draft QA Project Plan (QAPP) between p. 21 and p. 22. This sample collection/ laboratory receipt record is maintained by L.C. Metals (one copy is returned to Chemetco after laboratory receipt). Information on this form are transferred to a bound lab record book. The laboratory sample receipt records, laboratory analysis records and EP Extract solutions are under the direct control of Ms. Drumhiller, Supervisory Chemist, L.C. Metals. These procedures are acceptable; however, details of the lab analysis records will be discussed later in this report.

Soon after EP Extract preparation, sample aliquots are digested for cadmium and lead analysis per Method 3010, SW-846.

1. Observation: Sample aliquots are stored, as filtered, prior to aliquot digestion.

Recommendation: The laboratory should add 2 to 5 ml nitric acid per liter of extract immediately after extract filtration to minimize any metals precipitation. This is not specifically mentioned in Method 1310, but it is a common sample preservation practice. Samples should be acidified to pH < 2. This was discussed with L.C. Metals and Chemetco.

III. ANALYTICAL METHODOLOGY

L.C. Metals uses 1) the EP Extraction apparatus, described above, 2) digestion of extract sub-aliquots (50 ml) using Method 3010, SW-846, 3rd ed., and 3) flame atomic absorption for the measurement of cadmium and lead in the EP extracts, and 4) method of standard addition techniques for the quantitation of the 2 metals' concentrations. This is acceptable methodology specifically mandated by SW-846, 2nd and 3rd editions, for the EP Toxicity Test for these 2 metals.

The laboratory was having a certain difficulty in quantifying metals concentrations by the method of standard additions. Part of this is due to unclear specifications and details in SW846. Ten to 15% of past control solution measurements were biased unreasonably high. Also, a solution measurement and 2 spikes were being used for the method of standard additions. The following recommendations were made for the use of method of standard additions calculations.

A. Recommendations

1. Always use the linear part of the instrument calibrations curve. Dilute any large cadmium or lead sample concentration so that the linear range is used. If a non-linear part of the curve is used, inaccurate (biased-high) results commonly occur, to the detriment of Chemetco. One of the cadmium standard addition concentrations should be decreased, because it is too large.
2. Pick the sample spike concentrations to provide absorbance comparable to that of the sample absorbance.
3. Use a 4 point standard addition curve instead of a 3 point curve (SW-846 is unclear on the number of required additions). Four points are recommended based on our previous and past experience. The method of standard addition calculation should provide a linear regression plot with a correlation coefficient of 0.995 or greater for reasonably accurate concentrations to result.
4. Compare the unspiked metal concentration (determined from the instrument calibration curve) with the concentration calculated from the method of standard additions. The two values should be consistent, taking into account the slope of the two curves.
5. Most errors associated with imprecise method of standard addition calculations, as observed at L.C. Metals, work to the detriment of Chemetco.

IX. QUALITY ASSURANCE & DATA HANDLING

L.C. Metals has established appropriate quality control practices for the measurement of metals after the EP Extractions. Blanks and a control solution are digested with each set of sample extracts. The flame atomic absorption instrument is calibrated with several standards and is checked for calibration accuracy with standards and blanks throughout the analysis run. Each set of Chemetco samples analysis records are maintained in a file. These records include 1) calculations, 2) instrument hard copy read-outs of pH from the extraction pH controller, and 3) of absorbance from the flame atomic absorption instrument. The following recommendations were made and discussed with L.C. Metals and with Chemetco personnel in order to formalize and document the acceptable QA practices being done.

1. The accuracy of the pH controller system must be checked and documented at the end of each 24 hour extraction time period, (or more often if desired), in addition to the initial pH calibration.
2. A sample preparation log must be established to record dates and uniquely identify each solution digested (blank, duplicate, control, sample, diluted sample aliquots, etc.) by Method 3010.
3. The instrument read-out must identify each calibration standard (including zero concentration standards or instrument blanks) used for instrument calibration and identify each calibration standard or blank used to check calibration accuracy during a run. These calibration check standard and blank values must be recorded, and reviewed for acceptability, prior to re-zeroing of the instrument. A calibration check standard and blank must be recorded at the end of each set of sample analyses, and throughout the run as appropriate.
4. Analysis records for previous control solutions (carried through the digestion step) indicated generally accurate results at the EP Toxicity action levels; however, 10-15% of the results were biased high with errors of 25-75% reasonable control limits for these analyses (approximately 85-15% recovery). If control limits are exceeded, corrective action should be taken and analysis redone such as reanalysis for the extracts or redigestion of the EP Extracts.

cc: A. Jirka, DQAB
Attn: M. Long
D. Payne, CRL